INTERNATIONAL

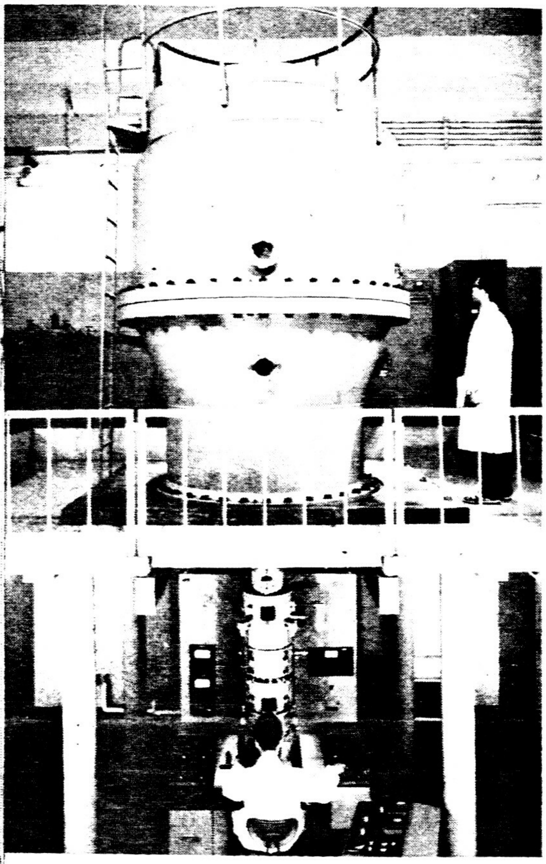
Electron Microscopy with Superconducting Lenses
By: H. Fernandez-Moran (HC) $1.00 (MF) .50

14 pages, code 1, CR-78745, cat. 14, NSG 441

N66 39339

Voltage, lenses aid electron microscopy

Developments push instruments closer to ultimate theoretical resolution of about 2 angstroms



FIRST MODEL. Operator sits at first model of 1 million volt electron microscope that the Japanese company Hitachi, Ltd., unveiled just last July

Two key developments—superhigh accelerating voltages (1 million volts and up) and superconducting lens systems—seem poised to lift the electron microscope to even greater heights of utility and push the instrument closer to its ultimate theoretical resolution of about 2 A. (The most sophisticated systems now give resolutions of about 10 A.) That was the outlook in Kyoto, Japan, early this month where some 1500 scientists from 36 countries gathered for the Sixth International Congress for Electron Microscopy.

The developments represent two somewhat different approaches to improving the design of electron microscopes. High voltage is aimed at increasing resolution as well as penetration. This means thicker specimens, a boon for both biological scientists and metallurgists. And it makes possible the study of living specimens.

Superconducting lenses operating at cryogenic temperatures give exceptional stability and high resolution, among other things. This means sharper electron micrographs at relatively low voltages. And it makes possible long exposures at low beam intensity, which produce fine detail even with relatively large fields.

Actually, the two approaches complement each other and future designs will likely incorporate features of both. Together, they should push electron microscopes closer to their theoretical capabilities.

But current design improvements should involve more than mere gadgetry. As one delegate to the Kyoto meeting put it: "The discussion has now been joined as to just what does this [high voltage systems, etc.] buy you?" With commercial 1 million volt models going for about $1 million, it's a fair question.

Of the two approaches, the movement to high voltage is furthest along, at least commercially. Three instruments of 1 million volts or more are now in use: one in France, two in Japan.

The French, pioneers in high voltage electron microscopy, since 1962 have been operating the world's largest unit at the Laboratoire d'Optique Electronique du C.N.R.S. at Toulouse. "We can reach 1.5 million volts and we work daily at 1.3 million volts," C.N.R.S. scientist Gaston Dupouy told C&EN in Kyoto.

"High voltage electron microscopes open up a new domain for the study of life," says Dr. Dupouy, one of the world's outstanding microscopists. High voltage instruments make it possible to see living cells at high magnification (50,000 ×, for example) and to follow the life cycles of organisms like bacteria, he adds.

Special chamber. At Toulouse, the French scientists use a specially built micro chamber to house bacteria for study by their big instrument. "It's a sort of flat or apartment for bacteria, about 0.1 cc. in volume, adjusted to the usual conditions of life," says Dr. Dupouy.

The "flat" has two small windows made of collodion thick enough to contain the atmosphere inside against the high vacuum of the instrument. The collodion windows pose no problem for the high energy electron beam used at Toulouse. Nor does the beam damage specimens.

For metallurgists and others studying solid specimens, high voltage microscopy means much greater penetration. "We can study and make images of films of metals about 50 times thicker than specimens used with commercial microscopes," says Dr. Dupouy.

What of the future? Dr. Dupouy

BIG ONE. French scientist Gaston Dupouy (left) tells colleague of the 1.5 million volt electron microscope at Toulouse feels 3 to 5 million volts may well be possible within the next five years.

But meanwhile, the Toulouse microscopists are devising ways to improve the performance of their present unit. At Kyoto, Dr. Dupouy described a simple screening device that eliminates nonscattered electrons. These electrons bear no information and serve only to blur the image. The result is dramatically sharpened micrographs.

But the French instrument is a laboratory model. And although the French and others in Europe have pioneered in high voltage electron microscopy, the Japanese are first with commercial models.

Two Japanese firms are now marketing 1 million volt machines priced at about $1 million in the U.S. Japan Electron Optics Laboratory Co. (JEOL) last April completed its first unit. And Hitachi, Ltd., unveiled its first model just last July (C&EN, July 25, page 39).

The two instruments bear similar specifications. JEOL's JEM-1000, for example, can generate 500, 750, or 1000 kv. The machine gives a resolution of "better than 10 A.," gives direct magnification of 500 × to 150,000 ×, and operates with a voltage stability of 2×10^{-5}/minute. Penetration with aluminum exceeds 5 microns. Hitachi specifications are not too different.

First thoughts. The original thinking behind the two units wasn't necessarily commercial. One JEOL executive says, "We built it [the JEM-1000] as a matter of prestige. But now it looks interesting [commercially]." Both firms, in fact, now say they have several prospective customers in tow.

JEOL has four "hot prospects": two in Britain, one in the U.S., and one in the U.S.S.R. Britain's atomic energy establishment at Harwell seems closest to dealing and likely will be JEOL's first customer. JEOL won't reveal the name of its potential buyer in the U.S. But a good bet is Georgia Tech, which is now planning a laboratory to house a million volt machine. Hitachi has also been active and says it has at least one good prospect in the U.S. Meanwhile, it has received an order from West Germany's Max Planck Institut für Hirnforschung-Neuroan, in Frankfurt, for a 650,000-volt unit.

Enthusiastic as the two firms are, the big units hardly represent a mass market, not at $1.00 a volt. Still, the machines generated considerable interest at the Kyoto conference, where both firms presented papers outlining basic structural details. And both firms hauled busloads of interested delegates to their plants for a firsthand look at the microscopes.

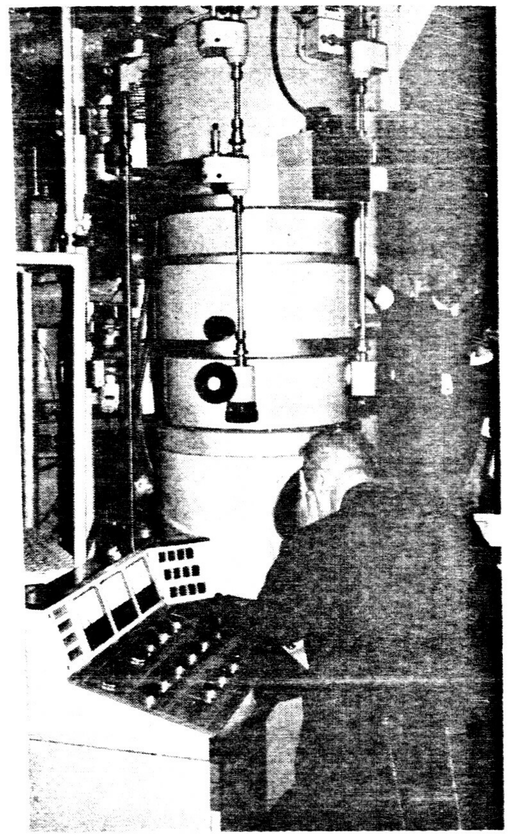
The second approach to improved microscope design, use of superconducting lenses, has been championed by Dr. Humberto Fernández-Morán and his colleagues at the University of Chicago's department of biophysics. Others are also now moving into the field, including Dr. Susumu Ozasa and others at Hitachi's central research laboratory, Dr. Benjamin M. Siegel and others at Cornell, and Dr. A. Laberrigue and others at the College de France, in Paris.

As Dr. Fernández-Morán puts it: "One way is to build high voltage machines. . . . But beyond a million volts or so, the lens becomes huge . . . the thing starts to become a monster. . . ." So Dr. Fernández-Morán tackled the problem from another direction by developing superconducting lens systems as well as working out some of the engineering problems that afflict existing electron microscopes. This has meant a prodigious R&D effort, the 42-year-old Venezuelan says.

"We have taken a systems approach," Dr. Fernández-Morán told the Kyoto conference. "We have worked not only on the lens, but on the specimen mounting, the power source, the vacuum system, circuitry, and so on."

The problems were many. One of them was incorporating superconducting solenoid lenses and related cryogenic equipment into an electron microscope system. Another was working out precise control and reproducible current settability for focusing superconducting lenses. Yet another was developing an adequate specimen mounting that prevents temperature drift. Another was stabilizing lens excitation current and accelerating voltage. And there were many more.

To solve these and other problems, Dr. Fernández-Morán and his colleagues approached the work from two angles. They worked out what he calls a cryo-electron microscope optical bench system. This system includes a modified air-core liquid helium Dewar with different types of niobium-zirconium solenoid lenses. The lenses operate at 4 to 32 kilogauss without pole pieces and with modified objective and objective projector pole pieces.



BRITISH GUEST. British guest peers into scope of JEOL's new 1 million volt electron microscope at the Japanese company's plant near Tokyo

U.S. GUEST. University of Chicago's Fernandez-Moran (right) greets colleagues at exhibit of his electron micrographs at Kyoto meeting

The other approach was to develop a superconducting objective lens in a specially designed liquid helium Dewar. This lens system can then be used as an integral part of a cryo-electron microscope. Or it can be used to replace the objective lens in a modified high-resolution commercial electron microscope. This lens system, designed by Westinghouse to Dr. Fernández-Morán's specification, includes niobium-zirconium main and vernier coils with superconducting stigmators, persistent current switches, and current control devices.

The effort seems to be paying off. Dr. Fernández-Morán said at Kyoto that he has produced exceptionally stable, high quality images of biological specimens, using a superconducting objective lens of niobium-zirconium at liquid helium temperature. In his most recent work, he says, he has attained resolutions of 10 to 20 A. at 4.2° K. using test specimens of stained catalase asbestos on carbon film and a superconducting objective lens without pole piece in persistent current mode at 75 kv.

High stability. In other work, Dr. Fernández-Morán, taking advantage of the high stability of superconducting lenses, has made exposures of from 30 seconds to several minutes using a low-intensity beam and high-resolution plates. In this way, he has been able to produce micrographs showing a wide field and fine detail "normally only discernible in the best high resolution micrographs at higher magnification."

Thus, though the problems are many, so are the advantages. In addition to achieving "superstability," Dr. Fernández-Morán says, his system also keeps contamination and specimen damage to a minimum, makes for optimum use of field emission, and embodies optimum conditions for both low and high voltage work, among other things.

Dr. Fernández-Morán's work with cryo-electron microscopes goes back to 1958, but he really got serious about it five years ago. "In the beginning, I was laughed at," he now says with a hint of pleasure. "People said it was too hard." He notes that work in the field has been mainly a U.S. development.

What is the outlook for cryo-electron microscopy? "A whole new optics is coming into being," says Dr. Fernández-Morán. "And it has implications for computers, too. The way is open for new approaches which are unique to superconductors as perfect diamagnetic materials. The real dividend to come will be when we can change configurations, use more efficient lenses, and so forth." But commercial models are still "a few years" off, he adds.